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(54) Masking a Substrate During Pack Aluminising

(57) A mixture for the partial masking of a metal surface during pack aluminising comprises a particulate material, such as zirconium silicate, which will form a non-gaseous reaction product with halides and sinter at aluminising temperatures to

provide a barrier impervious to molten aluminium. The particulate material is carried by an organic resin binder, such as polyvinyl alcohol, which will vapourise at aluminising temperatures leaving minimal residue. When particulate material used may be Ni or NiO with inert fillers such as ZrO₂ or Al₂O₃. The preferred binder is polyvinyl alcohol.

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SPECIFICATION

Improvements in or Relating to Aluminising

This invention relates to aluminising and in particular to a mixture for the partial masking of a metal surface whereby that surface may be selectively aluminised.

It is well known to improve the high temperature corrosion resistance of metals by aluminising. The surfaces of such metals are commonly aluminised by being embedded in a pack aluminising mixture containing aluminium particles, one or more halides and alumina. Such mixtures are, for instance described in U.K. Patent No. 1063412. It is sometimes necessary, however, to ensure that some portions of the surface of the metal are not aluminised. This may be necessary, for instance, if regions of the surface are required to be of very accurate dimensions. Aluminising almost invariably results in a small dimensional growth in the surface being aluminised and this may well be unacceptable in certain circumstances. A problem associated with surfaces which have been aluminised is that the aluminised coating is usually more brittle than the material of the surface. This makes regions of aluminised coatings susceptible to multiple cracking if those regions are subjected to comparatively minor strains at temperatures up to 700°C. It is usually desirable therefore to ensure that such regions are not aluminised.

It is an object of the present invention to provide a mixture for the partial masking of a metal surface whereby that surface may be selectively aluminised.

According to the present invention, a mixture suitable for the partial masking of a metal surface whereby that surface may be selectively aluminised comprises a particulate material adapted to form a non-gaseous reaction product with halides and to sinter at aluminising temperatures to provide a barrier impervious to molten aluminium, said particulate material being carried by an organic resin binder adapted to burn off or vapourise with minimal residue at aluminising temperatures.

When the partially masked workpiece is subjected to pack aluminising, the high temperatures involved (around 900°C) burn off or vapourise the organic resin binder to leave the particulate material on the workpiece surface. The particulate material then sinters to form a barrier which prevents aluminising of the masked areas of the workpiece.

When a metal surface is pack aluminised, aluminium is believed to reach the surface by two routes: by melting and flowing on to the surface and by reacting with halides present in the pack mixture to form gaseous aluminium halides which subsequently decompose on the surface to yield aluminium. After reaching the metal surface, the aluminium subsequently diffuses into it. We believe that the sintered particulate material present in mixtures in accordance with the present invention prevents aluminising by firstly

forming non-gaseous reaction products with gaseous halides and secondly providing a barrier which is impervious to molten aluminium.

Said particulate material may comprise zirconium silicate.

Said zirconium silicate may be less than 53 μm particle size.

Under certain conditions of aluminising, usually those in which the aluminising temperature exceeds 900°C and the pressure and concentration of halides are high, there is a tendency for the zirconium silicate to decompose and deposit silicon on the metal surface being aluminised. This is most noticeable at the boundary of the mask. Since such silicon could be absorbed by the metal surface and have possible detrimental effects upon its properties, we prefer to replace the zirconium silicate in such circumstances with finely divided particles of nickel or nickel oxide.

If finely divided particles of nickel or nickel oxide are used, we have found that when the particles sinter at aluminising temperatures, they sometimes fuse to the surface of the metal being aluminised making their removal extremely difficult. In order to prevent this we add an inert filler to the mixture, the inert filler constituting up to 50% by weight of the dry mixture in the case of nickel particles and up to 20% by weight of the dry mixture in the case of nickel oxide particles.

We also prefer to add an inert filler material to mixtures containing zirconium silicate if the surface to be masked is of a particularly complex configuration. When masking such surfaces, we have found that zirconium silicate without an inert filler tends to flow away from the sharp edges to provide a coating of uneven depth. This is undesirable since there is a possibility of the masking failing in areas where it is of insufficient depth. If an inert filler is added to zirconium silicate mixture, we have found that an acceptably even coating thickness is achieved.

The inert material may be calcined or fused aluminium oxide although in the case of mixtures containing nickel or nickel oxide particles, we prefer to use zirconium oxide. Moreover when zirconium oxide is used, we have found that it is most effective when present as a mixture of zirconium oxide particles of different sizes. Thus we have found a particularly useful zirconium oxide particle mixture to contain 50% by weight of zirconium oxide particles of less than 10 μm particle size with the remainder zirconium oxide particles of less than 53 μm particle size. Nickel and nickel oxide particles of less than 75 μm and 45 μm particle sizes respectively are particularly effective when mixed with zirconium oxide particles of the previously mentioned particle size ranges.

The organic resin binder may be polyvinyl alcohol.

The particulate material, with or without an inert filler material, and polyvinyl alcohol may be mixed together in the presence of water so as to form a slurry. The slurry may then be applied by, for instance, brushing to form a mask on those

areas of a metal surface which are to be protected from aluminising. It will be appreciated however that other techniques of application such as dipping or spraying may be usefully employed in certain circumstances. After application, the coating is allowed to air dry although this may be speeded up by slight warming.

The amounts of organic resin binder and water necessary to achieve an effective mixture depend on the method of application to the metal surface to be protected from aluminising. Simple experiments to determine the most effective mixture for the method of application chosen may be easily performed before the mixture is actually applied to the metal surface concerned.

After aluminising, we have found that the masking may be removed from the metal surface by brushing to leave a bright non-aluminised surface. Under certain circumstances, however, for instance in the masking of particularly inaccessible regions of a workpiece, it may be desirable to aid the removal of the masking after aluminising. This may be achieved by coating those areas of the metal surface which are to be masked with a thermally decomposable resin, such as methyl methacrylate, prior to application of the particulate material slurry.

The invention is illustrated with reference to the accompanying examples:—

30 Example 1

The aerofoil portion of a turbine blade manufactured from a nickel base superalloy known as Nimonic 115 was divided into four zones by strips of paper masking tape. Three layers of methyl methacrylate resin were then applied by brushing to the exposed zones of the aerofoil in order to act as a parting agent.

The exposed zones of the aerofoil were then coated with a slurry of zirconium silicate of 53 μm particle size in polyvinyl alcohol and water by brushing. Subsequent layers of the slurry were brushed on to the aerofoil after air drying until the four exposed zones were respectively coated with one, two, three and four coatings of the slurry.

After removal of the paper masking tape, the turbine blade was pack aluminised for eight hours at a maximum component temperature of $915^{\circ}\text{C} \pm 10^{\circ}\text{C}$ in the following pack mixture:

	by weight
50 Particulate Aluminium	1.3—1.7%
Ammonia Chloride	0.1—0.2%
Sodium Fluoride	0.8—1.2%
Potassium Bifluoride	0.05—0.15%
Calcined Alumina	Remainder

After aluminising, the sintered zirconium silicate was removed from the turbine blade using a stiff fibre brush. The effectiveness of the zirconium silicate in preventing aluminising was judged on the basis of the appearance of the aerofoil surface. Thus a pink or blue colouration is indicative of aluminising penetration whilst a straw gold colour is indicative of the successful

prevention of aluminising. We have substantiated these colour indications by the examination of similar aluminised and non-aluminised surfaces at approximately 500 \times magnification.

The following colours were observed on the aerofoil:—

	1 Coat	2 Coats	3 Coats	4 Coats
70	Blue-Gold	Gold	Gold	Gold

Thus two coats of a zirconium silicate slurry were effective in preventing aluminising.

Example 2

Example 1 was repeated replacing the zirconium silicate slurry with a slurry containing:—

25%	part by weight zirconium oxide	53 μm particle size.
25%	part by weight zirconium oxide	10 μm particle size.
80	50% parts by weight nickel powder	75 μm particle size.

and sufficient polyvinyl alcohol and water to provide a consistency suitable for brush application.

After aluminising, the sintered zirconium oxide and nickel was removed from the turbine blade using a stiff brush. The following colours were observed on the aerofoil:—

	1 Coat	2 Coats	3 Coats	4 Coats
90	Blue-Gold	Gold	Gold	Gold

Thus two coats of a zirconium oxide/nickel slurry were effective in preventing aluminising.

Example 3

Example 1 was repeated replacing the zirconium silicate slurry with a slurry containing:—

10%	part by weight zirconium oxide	53 μm particle size.
100	10% part by weight zirconium oxide	10 μm particle size.
80%	parts by weight nickel oxide powder	45 μm particle size.

and sufficient polyvinyl alcohol and water to provide a consistency suitable for brush application.

After aluminising the sintered zirconium oxide and nickel oxide was removed from the aerofoil blade using a stiff fibre brush. The following colours were observed on the aerofoil:—

	1 Coat	2 Coats	3 Coats	4 Coats
110	Blue	Blue-Gold	Gold	Gold

Thus three coats of a zirconium oxide/nickel oxide slurry were effective in preventing aluminising.

Claims

1. A mixture suitable for the partial masking of a metal surface whereby that surface may be selectively aluminised comprising a particulate material adapted to form a non-gaseous reaction product with halides and to sinter at aluminising temperatures to provide a barrier impervious to molten aluminium, said particulate material being carried by an organic resin binder adapted to burn off or vapourise with minimal residue at aluminising temperatures.
2. A mixture suitable for the partial masking of a metal surface as claimed in claim 1 wherein said particulate material comprises zirconium silicate.
3. A mixture suitable for the partial masking of a metal surface as claimed in claim 2 wherein said zirconium silicate is less than $53\text{ }\mu\text{m}$ particle size.
4. A mixture suitable for the partial masking of a metal surface as claimed in claim 2 or claim 3 wherein said mixture also comprises an inert filler material.
5. A mixture suitable for the partial masking of a metal surface as claimed in claim 1 wherein said particulate material comprises finely divided particles of nickel and an inert filler material.
6. A mixture suitable for the partial masking of a metal surface as claimed in claim 4 wherein said inert filler constitutes up to 50% by weight of the dry weight of the mixture.
7. A mixture suitable for the partial masking of a metal surface as claimed in claim 5 or claim 6 wherein said finely divided particles of nickel are less than $75\text{ }\mu\text{m}$ particle size.
8. A mixture suitable for the partial masking of a metal surface as claimed in claim 1 wherein said particulate material comprises finely divided particles of nickel oxide and an inert filler material.
9. A mixture suitable for the partial masking of a metal surface as claimed in claim 8 wherein said inert filler material constitutes up to 20% by weight of the dry weight of the mixture..
10. A mixture suitable for the partial masking of a metal surface as claimed in claim 8 or claim 9 wherein said finely divided particles of nickel oxide are less than $45\text{ }\mu\text{m}$ particle size.
11. A mixture suitable for the partial masking of a metal surface as claimed in any one of claims 5 to 10 wherein said inert filler material is particulate zirconium oxide.
12. A mixture suitable for the partial masking of a metal surface as claimed in claim 11 wherein 50% of said particulate zirconium oxide is less than $10\text{ }\mu\text{m}$ particle size with the remainder less than $53\text{ }\mu\text{m}$ particle size.
13. A mixture suitable for the partial masking of a metal surface as claimed in any preceding claim wherein said organic resin binder is polyvinyl alcohol.
14. A mixture suitable for the partial masking of a metal surface substantially as hereinbefore described with reference to the accompanying examples.